

# REPORT DOCUMENTATION PAGE

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13. ABSTRACT (Maximum 200 Words)  The objective of this grant was the acquisition of a hyperthermal atomic oxygen (AO) source that is of critical importance to a Multi-University Research Initiative (MURI) on the fundamental mechanisms of materials degradation in the low earth orbit (LEO). ("Simulations of Fundamental Degradation Process in Space Environments", PI: J. C. Yang, MURI Contract #: F49620-01-1-0336). A Physical Sciences, Inc. (PSI) FAST™ (Fast Atom Sample Tester) source was specially designed for portability with an ultra-high vacuum exposure chamber and delivered to University of Pittsburgh in October 2003. The portability of this FAST™ AO source allows the utilization of unique experimental tools located at different sites. This system has now been improved so that the best base vacuum is $4 \times 10^{-9}$ torr. A room temperature sample stage has been built, a dual head quartz crystal monitor (QCM) system has been installed for <i>in situ</i> weight loss. A sensitive Mettler balance was purchased in order to measure total weight changes. All of these items have been installed and working since May 2004. Present experiments are being performed on Si, Al, Ag and Au single crystals and thin films.			
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**Defense University Research Instrumentation Program  
DURIP**

**ACQUISITION OF HYPERTHERMAL ATOMIC OXYGEN SOURCE**

**FINAL REPORT  
AUGUST 11, 2004**

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**DURIP Grant Number: F49620-02-1- 0198**

**Program Manger:**

**Lt. Col. Paul Trulove (past), AFOSR**

**Maj. Jennifer Gresham, PhD (present, since 8/2004), AFOSR**

**Start Date: May 1, 2002**

## A. OBJECTIVES

The objective of this grant was the acquisition of a portable hyperthermal atomic oxygen (AO) source that is of critical importance to a Multi-University Research Initiative (MURI) on the fundamental mechanisms of materials degradation in the low earth orbit (LEO). ("Simulations of Fundamental Degradation Process in Space Environments", PI: J. C. Yang, MURI Contract #: F49620-01-1-0336, Program Manager: Lt. Col. Paul Trulove, AFOSR, present program manger: Maj. J. Gresham, AFOSR). Hyperthermal atomic oxygen is the primary species in the low earth orbit that rapidly degrades materials used in space vehicles. The Physical Sciences, Inc. (PSI) FAST<sup>TM</sup> (Fast Atom Sample Tester) sources are the only hyperthermal atomic oxygen sources capable of continuous operation at both the appropriate orbital velocity and high flux. This source employs a laser-supported detonation wave heating of pure molecular oxygen to generate atomic oxygen continuously with kinetic energy of 5 eV with high flux. Physical Sciences, Inc. (PSI) (contact: Bob Krech) specially designed and manufactured a FAST<sup>TM</sup> AO source, that was portable (on wheels), contained an ultra-high vacuum (UHV) chamber, and capable of very low dose for monolayer oxidation studies. This FAST<sup>TM</sup> AO source was delivered to University of Pittsburgh in October 2003.

The portability of this FAST<sup>TM</sup> AO source allows the utilization of unique experimental tools located at different sites. Specifically, ultra-high vacuum scanning transmission electron microscopy (UHV-STEM) and Surface Science Center, located at University of Pittsburgh, and synchrotron X-ray Diffraction (XRD), located at Brookhaven National Laboratory and Argonne National Laboratory.

## A. STATUS OF EFFORT

The negotiation between PSI and the University of Pittsburgh took several months to clarify issues such as warranty (PSI finally agreed to a 1 year warranty), training, final demonstrated performance, and delivery costs. The Pittsburgh FAST<sup>TM</sup> AO source was designed, built, and delivered in October 2003 by Bob Krech, where installation and training was conducted at Pittsburgh. The Pittsburgh FAST<sup>TM</sup> 5 eV atomic oxygen system includes a cryopumped stainless steel vacuum test chamber with a pulsed nozzle assembly, a radiometric photomultiplier based beam velocity measurement system, a laser beam focusing system, control electronics, and a CO<sub>2</sub> laser. The primary FAST<sup>TM</sup> AO Source specifications are listed in Table 1.

Fast O-Beam Properties	
Velocity	8 km/s $\pm$ 15% (5 to 12 km/s range)
Fluence	$\sim 10^{18}$ O-atoms/pulse, 3 Hz
Composition	>80% oxygen atoms
Size	Expandable to >1,000 cm <sup>2</sup> area
Charge content	<1% ions (controllable by pseudo-Helmholtz coils)
Metastable content	O( <sup>1</sup> D) concentration <0.4%
Temperature	T = 300 K
VUV/UV content	One photon per 10 <sup>4</sup> O atoms (similar to LEO)

Table 1. Summary highlights the properties of the oxygen beam.

The base vacuum of the sample chamber of the AO source after delivery was about 10<sup>-5</sup> torr, in part due to a complication with the turbo pump control system. The Pittsburgh FAST<sup>TM</sup> AO source was also missing some items that were necessary to use it, such as a sample stage, bake-out system (since it was designed to be UHV), and a system to measure the fluence and species within the beam. The vacuum system and monitoring system has now been improved so that the best base vacuum is now 4x10<sup>-9</sup> torr. Furthermore, a room temperature sample stage has been built, a dual head quartz crystal

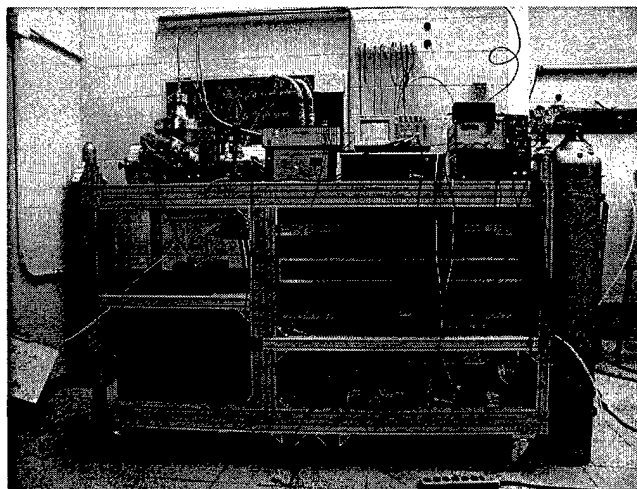
monitor (QCM) system has been designed and purchased and a very sensitive Mettler balance has been purchased to measure weight changes. The QCM system was designed to measure weight loss of thin films, such as Ag or C, that are standard materials to calibrate the AO fluence for very small AO doses. The Mettler balance was purchased in order to measure weight changes of Kapton, which is a standard witness material to determine total AO fluence. All of these items have been installed and working since May 2004. Table 2 is the list of items that were purchased, including the original cost of the FAST<sup>TM</sup> AO source, that was covered by the DURIP award:

No.	P.O.# Date	Category	Description	Vendor	Prices (\$)
1	94863 10/28/2003	FAST AO	Compact style.	PSI	350,000
2		Power Supply Cable	For AO source power supply, NEMA type	Made at Pitt	200
3	456202	4 Regulators and gases	O <sub>2</sub> ,	With regulators \$275each Penn Oxygen	446
			N <sub>2</sub>		311
			Laser mix75		475
			He		402
4	467861	Hazard gas detector	Model HIC, for CO, etc.	M <sup>c</sup> . Master-Carr	177
5		Sample stage	Room temperature	Made in Pitt machine shop	200
6	473267	High vacuum gauge with controller	MKS 943, 10 <sup>-10</sup> to 10 <sup>-2</sup> Torr range	Kurt J. Lesker Company	1,095
7	490371 05/06/2004	QCM(quartz crystal microbalance) sensor with RQCM controller	Dual bake-able sensor heads, RQCM controller with sensor crystals	Maxtek, Inc.	7,484
8	494735	PC computer	For RQCM	Dell	1,336
9	509179	Internal bake-out heater (IR halogen quartz lamp) with controller	IR-123-120	Huntington Mechanical Labs. Inc.	1,686
10	105073 02/16/2004	Microbalance	Mettler Toledo MX5, Readability 0.01 mg	Fisher Sci.	11,185

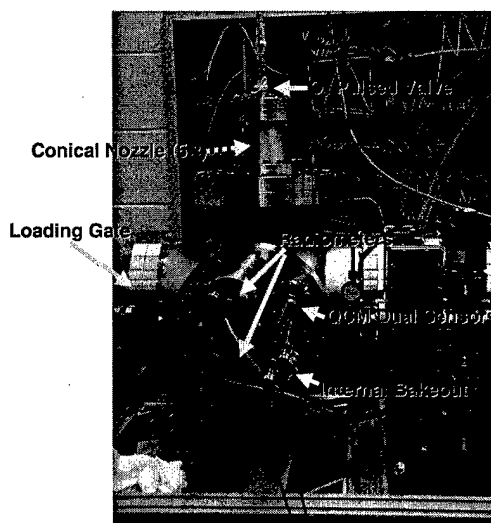
<b>TOTAL:</b>	<b>\$374,997</b>
DURIP award:	\$350,000
Pitt contribution through MURI-AFOSR award:	\$24,997

Table 2: Summary of items and costs for the Pittsburgh FAST AO source.

The fluence as determined by both the weight loss of the Kapton witness sample and Ag weight gain as monitored by the QCM system was approximately  $4 \times 10^{19}$  AO/cm<sup>2</sup> and  $2 \times 10^{19}$  AO/cm<sup>2</sup>, respectively for 50,000 shots or approximate  $6 \times 10^{14}$  AO/cm<sup>2</sup>/shot where the distance between the nozzle and sample stage was 30 cm. This dose can be altered by varying the distance between the sample and the nozzle. Figure 1 is an image of the Pittsburgh FAST AO source, and Figure 2 is a close-up image of the ultra-high vacuum chamber for this source.



*Figure 1: Pittsburgh FAST AO source.*



*Figure 2: Image of the UHV chamber of the Pittsburgh FAST AO source.*

## C. ACCOMPLISHMENTS

Thin films of Ag, Al and Au were coated onto the quartz crystal and the weight change was monitored by the QCM system. Au revealed no weight change, whereas Al showed some weight increase and Ag showed a significant weight increase (Figure 3). From previous electron microscopy investigations, it is known that the "oxide scale" on Ag due to AO exposure is mostly polycrystalline, thereby demonstrating that Ag does not oxidize but continuously absorbs oxygen, which explains the initial linear increase of weight of the Ag when exposed to atomic oxygen.

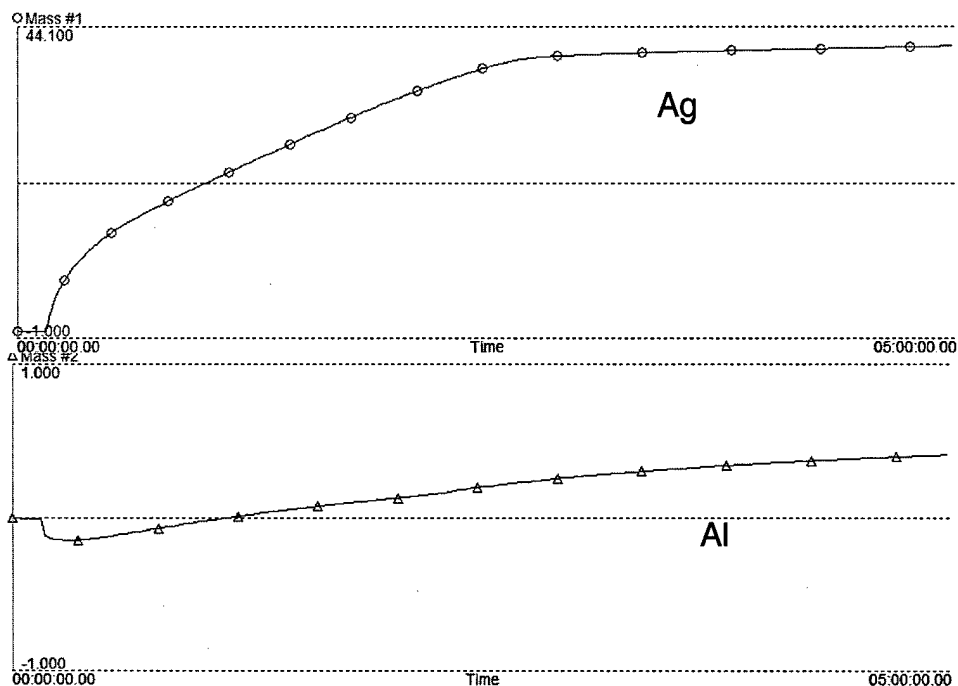


Figure 3: Weight gain of Ag and Al thin films when exposed to hyperthermal atomic oxygen within the Pittsburgh FAST AO source, as monitored by a dual-head quartz crystal monitor system. The total AO fluence is calibrated, using both a Kapton witness sample and Ag film, to be  $3 \times 10^{19}$  AO/cm<sup>2</sup> for 50,000 shots of the CO<sub>2</sub> laser. Silver shows a rapid increase in weight, whereas aluminum shows only a little increase in weight.

#### D. PERSONNEL

Judith Yang, Associate Professor, Materials Science and Engineering, University of Pittsburgh

Ian Robinson, Professor, Physics, University of Illinois at Urbana-Champaign

John T. Yates, Jr., Professor, Chemistry, University of Pittsburgh

Long Li, Research Associate, Materials Science and Engineering, University of Pittsburgh

Ross Harder, Post-doc, Physics, University of Illinois at Urbana-Champaign

Maja Kisa, Graduate Student, Materials Science and Engineering, University of Pittsburgh

## **E. PUBLICATIONS**

None to report at this time.

## **F. INTERACTIONS**

### **F. 1. Conference Presentations**

"Different Amorphous Oxide Structures Formed by Oxidation of Si and Al in Reactive Oxygen Species", Invited Talk, Speaker: Judith Yang, Microscopy and Microanalysis, 4 August 2004, Savannah, GA.

### **F. 2. Consultative Function**

1. Comparison between atomic oxygen sources at Montana State University and at University of Pittsburgh.
2. Development of Materials International Space Station Experiment (MISSE) 6 mission (includes AFOSR and Boeing funding) where identical samples will be exposed to the atomic oxygen in the low earth orbit and compared to the FAST AO source to determine how well the ground-based AO source compares to the space environment.

## **G. PATENTS**

None to report at this time.

## **H. HONORS & AWARDS**

Ian Robinson (Physics, University of Illinois at Urbana-Champaign) is a Fellow of the American Physical Society and the 2000 winner of the B. E. Warren Diffraction Physics Award of the American Crystallographic Association.

John T. Yates, Jr. is a R. K. Mellon Professor of Chemistry, University of Pittsburgh since 1981. He is a Member of the National Academy of Sciences (1996), the recipient of the von Humboldt Senior Scholar (1995-1997) and the J. Linnett Professorship from Cambridge University (2000).

Judith C. Yang (Materials Science and Engineering, University of Pittsburgh) is the 2002 recipient of a NSF Career Award on fundamental oxidation investigations.